NRL Report 6141

Electron Probe Microanalysis of the Diffusion Systems β-Titanium-Vanadium, β-Titanium-Niobium, and Chromium-Vanadium

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ABSTRACT

The diffusions of niobium and vanadium into pure β-titanium at 1100°C have been studied. Concentration versus distance curves for these systems are similar to each other but are skewed and different in shape from the Gaussians expected in self-diffusion. At temperatures between 1050°C and 1200°C the curves for chromium-vanadium diffusion do approximate Gaussians.

Diffusion coefficients for all three systems have been calculated by Matano's method at elemental concentrations from 5 atom-percent to 95 atom-percent. Logarithms of the diffusion coefficients D for β-titanium with both niobium and vanadium follow a reasonable straight-line relationship with the atom-percent of the solute element. The D values range from 3.5×10^{-12} cm^2/sec at 95 atom-percent Nb to 7.0×10^{-10} cm^2/sec at 5 atom-percent Nb, and from 4.5×10^{-11} cm^2/sec at 95 atom-percent V to 5.0×10^{-9} cm^2/sec at 5 atom-percent V. Calculated D values for Cr-V at 1050°C, 1100°C, and 1200°C are 4.5×10^{-14}, 1.3×10^{-13}, and 5.6×10^{-12} cm^2/sec respectively. A frequency factor, D_0, of 6 ± 4 cm^2/sec and an activation energy, Q, of 71 ± 4 kcal/g atom were also calculated for Cr-V.

PROBLEM STATUS

This is a final report on one phase of the problem. Work is continuing on other phases.

AUTHORIZATION

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β-TITAMIUM-NIOBIUM, AND CHROMIUM-VANADIUM

INTRODUCTION

The diffusion experiments reported in this report are a continuation of the electron probe investigations of binary diffusion and phase phenomena reported earlier by this laboratory (1). Diffusion coefficients are reported at a number of concentrations across each diffusion couple from 5 atom-percent to 95 atom-percent. These results enlarge somewhat on the excellent work of Peart and Tomlin (2), in which radioactive tracer techniques were utilized to study the low-level diffusions of elements into β-titanium.

The obvious advantages of electron probe microanalysis when applied to narrow, friable areas have been well delineated in the published literature and will not be treated here other than to say that for these particular diffusion studies the lack of suitable isotopes of either titanium or vanadium precludes the use of tracer techniques on these elements and necessitates some other type of quantitative chemical analysis across such diffusion zones.

The prime object of this report is to supply information which will be of value in such practical engineering applications as bonding or cladding techniques and which may be of significance in diffusion theory as well.

EXPERIMENTAL METHODS AND MATERIALS

The metals used were the following:

Chromium - 0.005-inch electroplate from chromic acid-sulphuric acid solution.

Vanadium - 0.125-inch plate of 99.9% purity obtained from The Vanadium Corporation of America.

Titanium - 0.125-inch plate of 99.7% purity, MST Grade 40 having 0.04%C and 0.25% Fe obtained from the Mallory-Sharon Metals Corporation.

Niobium - 0.125-inch plate of 99.95% purity obtained from Wah Chang Corp.

A number of 0.25 × 0.25 inch platelets of each metal were cut from the respective plates and given a good, flat metallographic polish on one face. To form the diffusion couples an appropriate pair of platelets, say titanium and niobium, was lightly spot welded under helium. A Pt/Pt-13%Rh thermocouple was then spot welded under helium to the couple, which was then placed in a small tungsten-wound quartz-tube furnace contained in a vacuum chamber. All diffusion heating was done at a pressure of less than 5×10^{-5} torr, and the temperature was controlled to ±2°C at temperatures between 1050° and 1200°C with a Leeds and Northrup controller-recorder actuated by the couple-attached thermocouple. At the completion of the diffusion time the specimen was cooled (at 250°C per minute in vacuum) to room temperature, removed from the furnace, mounted in Bakelite, and sectioned across the diffusion zone at a point well away from the couple edge. The specimen was then prepared for electron probe microanalysis by polishing the cross section smooth and flat using Struers DP-diamond paste, types A through C consecutively, on Struers DP-cloths, DUR and MOL. No etchants were applied to the diffusion couples prior to electron probe analysis.

Electron probe analysis was performed using 26-kev electrons focused to a 1-micron-diameter spot. The K_α x-ray intensities for each of the elements of interest in each diffusion couple were measured simultaneously using curved lithium fluoride crystals as wavelength discriminators and Geiger tubes as detectors. Birks' calculation technique was utilized to convert measured x-ray intensities to weight percents (3).

The wide βTi-Nb and βTi-V couples were analyzed at discrete spots, spaced 5 microns apart, for the entire width of the couple, while the narrower Cr-V couple was analyzed by continuous scan at a rate of 2 microns per minute.

Visual examinations at 500X prior to electron probe analysis did not disclose any evidence of voiding, contaminant inclusion, or precipitation in any diffusion zone. The extent and regularity of each zone was checked at a number of points by electron probe analysis and no intergranular diffusion or penetration was noted.

RESULTS

At temperatures above 900°C each of the elements chromium, vanadium, titanium, and niobium are in the bcc form designated as the A_2(W) structure type (4). β-titanium and niobium are of nearly the same atomic size, differing by only 0.5%. Chromium and vanadium are smaller, differing from β-titanium by 12% and 8% respectively and from each other by 5% (4). Tracer data on the β-titanium-niobium system has been reported by Peart and Tomlin who assumed that niobium should be analogous to vanadium in diffusion with β-titanium and expected that niobium diffusion in β-titanium and β-titanium self-diffusion should be in close agreement. These points will be discussed later in this section.

The pertinent features of our various diffusion couples are summarized in Table 1. Diffusion times were chosen strictly on the basis of operational convenience, since the width of a zone is rather unimportant in electron probe analysis. The widths of the zones were determined by electron probe technique and represent the measured distances between the closest points of approach of the pure elements after diffusion.

Table 1
Pertinent Features of the Diffusion Couples

Couple	Temperature (°C)	Time (hr)	Width of Zone	
			(Microns)	(Inches)
Cr-V	1050	72	9	3.5×10^{-4}
Cr-V	1100	93	24	9.5×10^{-4}
Cr-V	1200	72	115	4.5×10^{-3}
βTi-V	1100	48	1000	3.7×10^{-2}
βTi-Nb	1100	24	250	9.8×10^{-3}

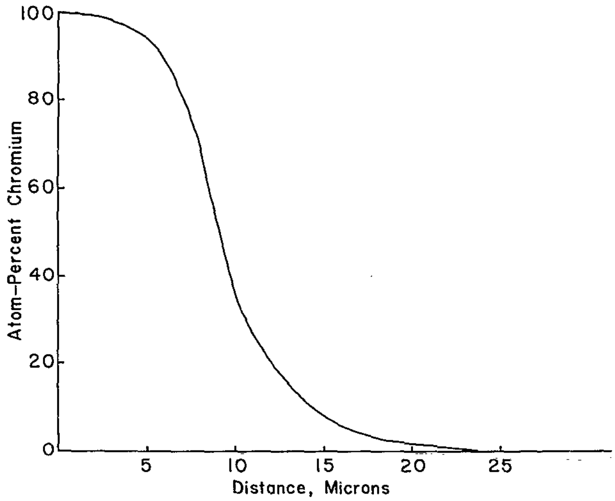

Fig. 1 - Typical concentration vs distance curve for Cr-V couple diffused at 1100°C for 93 hours

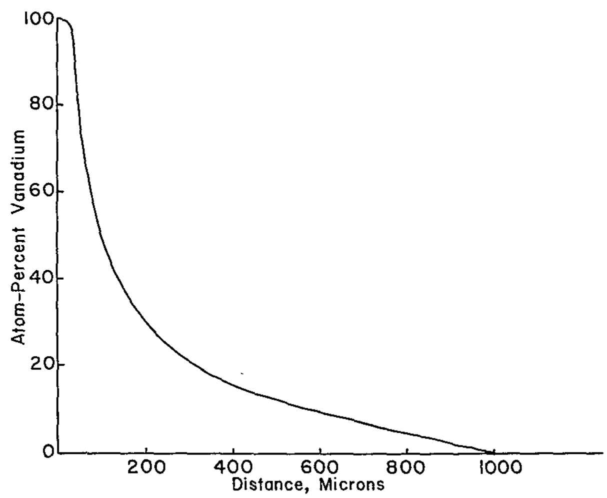

Fig. 2 - Typical concentration vs distance curve for βTi-V couple diffused at 1100°C for 48 hours

Figures 1 and 2 clearly illustrate the differences in diffusion zone shapes encountered during these experiments. The nearly Gaussian shape of the 1100°C Cr-V graph in Fig. 1 was typical also of those formed at 1050°C and 1200°C. Skewing of the β-titanium diffusion curves was quite apparent, with β-titanium acting as the solvent in each instance; the βTi-V curve is shown in Fig. 2.

Now, if self-diffusion, or its analog but involving in fact two different elements, does occur in a system, then, since each element is diffusing into the other at the same rate, a true Gaussian shape must occur in the curve of concentration vs distance. Also, in such a case, the diffusion rate must be independent of concentration of either of the diffusants. A skewed or asymmetric concentration vs distance curve represents a faster rate of diffusion of one element into the other than vice versa. Here there will be a definite dependence of diffusion rate on the concentration of the diffuser (solute), and this rate will be highest at the lower solute concentration for curves similar to that of βTi-V shown in Fig. 2.

The assumption of Peart and Tomlin that the diffusions of niobium and vanadium into β-titanium should be similar to one another appears to be correct. However, their further premise that niobium diffusion into β-titanium must be analogous to β-titanium self-diffusion seems to be in error. This finding is further substantiated by the plots shown in Fig. 3.

Diffusion coefficients were calculated for each system by Matano's method. When the logs of such values are plotted as in Fig. 3 the concentration dependence of the diffusion rate is easily noted. If a true self-diffusion system were so represented, the diffusion rates would be constant with respect to composition and consequently there would be no slope to the line. In practice, where two different elements are involved, self-diffusion may only be approximated, and then the slope of such a line must be taken as evidence for a mode of diffusion different from that of self-diffusion. Where many binary systems are involved, the slope, together with the average diffusion rate, may be used as a criteria for ordering the various systems with respect to some self-diffusion system at a particular temperature.

In Fig. 3 it is immediately apparent that for the real systems of βTi-V at 1100°C and Cr-V at both 1050°C and 1200°C straight lines are the best representation of the plotted data. Since the diffusion mode of Cr-V would not reasonably be expected to change at 1100°C, a best straight line is assumed to represent this data also, even though the data points show a slight undulation about the line. Similarly, in the case of the βTi-Nb there is a slight undulation about a best straight line; however, the line serves to indicate the general slope. It should be pointed out that except in the case of self-diffusion there is no requirement that such plots must be true straight lines, in fact, such an occurrence with an actual diffusion system would probably be a fortuitous event.

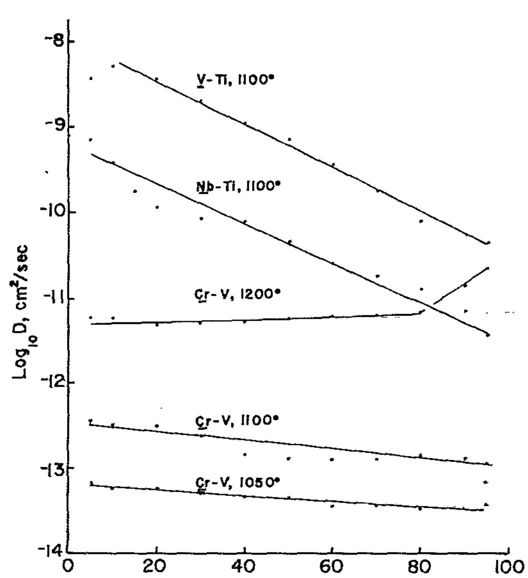

Fig. 3 - A composite graphing of the diffusion coefficients (D) obtained from this research. All values of D were calculated using Matano's method.

Table 2
βTi-Nb Diffusion at 1100°C

Atom-Percent Nb	Diffusion Coefficients (cm^2/sec)	
	NRL	Peart and Tomlin
5	7.0×10^{-10}	2.0×10^{-9}
10	3.7×10^{-10}	1.1×10^{-9}
15	1.76×10^{-10}	7.9×10^{-10}
20	1.16×10^{-10}	

A comparison of 1100°C βTi-Nb values with those of Peart and Tomlin is presented in Table 2. Considering the differences in calculation procedures and assumptions relating to the data (Peart and Tomlin had only the low concentration end of the total diffusion curve) the agreement is reasonably good. If, on the basis of our data in the 0 to 30 atom-percent niobium range, the total diffusion curve would be assumed to be Gaussian, then our calculated diffusion coefficients would double and very closely approximate Peart and Tomlin's results.

Only 1100°C diffusions were carried out on the β-titanium systems; however a moderate range of temperatures was covered in the Cr-V couples and thus made it possible to calculate a frequency factor (D_0) and activation energy (Q) for this system. Using the general formula $D = D_0 \exp(-Q/RT)$ the values $D_0 = 6 \pm 4$ cm^2/sec and $Q = 71 \pm 4$ kcal/g-atom were found.

The Q value is comparable to that reported by Hagel (5) and Bokshtein et al. (6) for self-diffusion in chromium. This figure is thought to be reasonably correct. However, due to the short temperature range and long extrapolation of the data, the D_0 value is rather unprecise.

CONCLUSION

Investigations of the diffusions of vanadium and niobium with β-titanium at 1100°C have shown that β-titanium is the solvent in each instance. Variation-of-concentration vs distance curves for these binaries are strongly skewed, thus evidencing strong concentration dependence of the diffusion rates and contradicting any assumption that βTi-Nb diffusion might be analogous to βTi self-diffusion. Logarithms of the diffusion coefficients D for βTi with both niobium and vanadium follow a reasonable straight-line relationship with the atom-percent of the solute element. The D values range from 3.5×10^{-12} cm^2/sec at 95 atom-percent Nb to 7.0×10^{-10} cm^2/sec at 5 atom-percent Nb, and 4.5×10^{-11} cm^2/sec at 95 atom-percent V to 5×10^{-9} cm^2/sec at 5 atom-percent V.

A short temperature range has been covered in the Cr-V system. D values were found to be approximately 4.5×10^{-14} cm^2/sec at 1050°C, approximately 1.3×10^{-13} cm^2/sec at 1100°C, and approximately 5.6×10^{-12} cm^2/sec at 1200°C. A frequency factor D_0 (6 ± 4 cm^2/sec) and an activation energy Q (71 ± 4 kcal/g-atom) were calculated for this system.

Although all three binary systems do form continuous series of bcc solutions, there are marked differences in diffusion zone characteristics which cannot be accounted for simply on the basis of atomic size.

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Diffusion coefficients for all three systems have been calculated by Matano's method at elemental concentrations from 5 atom-percent to 95 atom-percent.

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